Sergio Petrozzi

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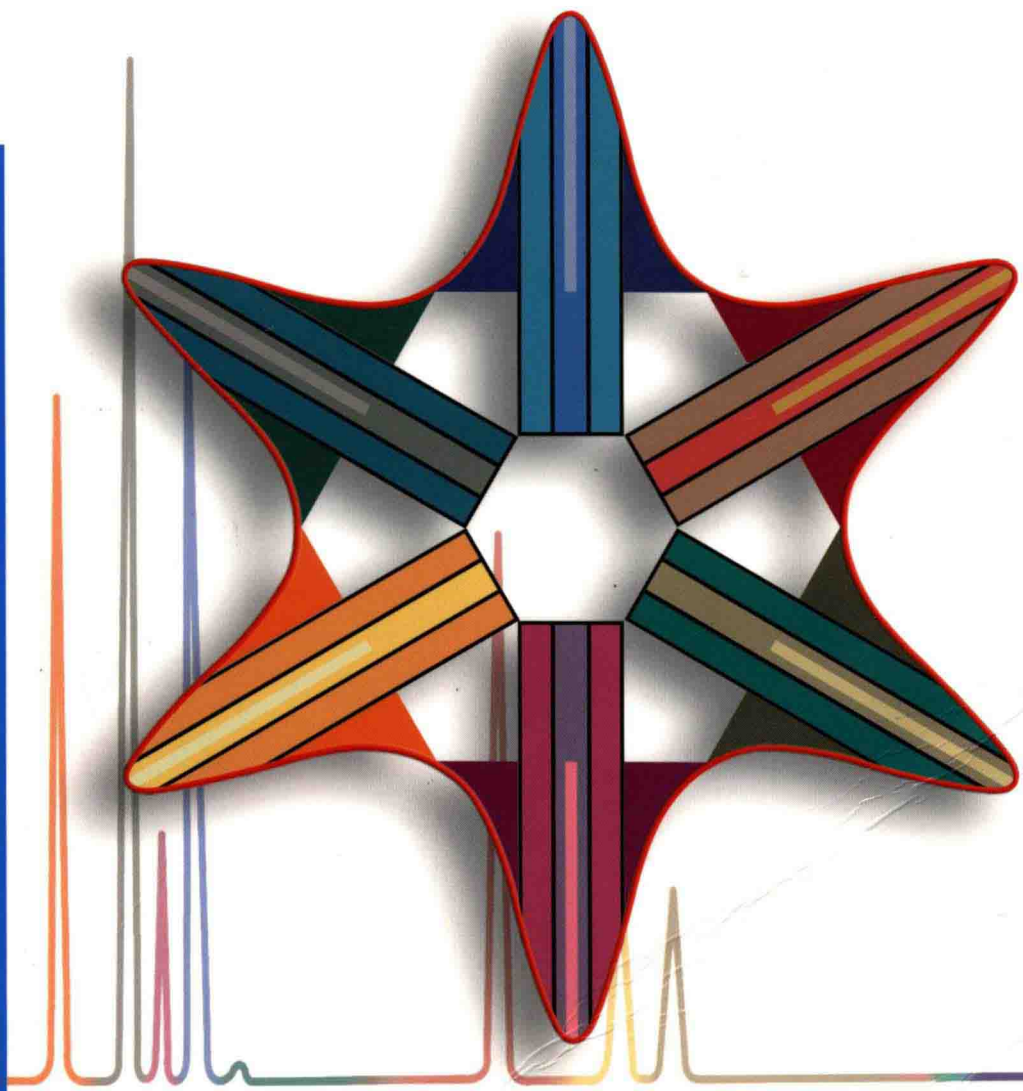
# Practical Instrumental Analysis

## Methods, Quality Assurance and Laboratory Management



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## Methods, Quality Assurance and Laboratory Management




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## Preface to the German Edition

An integral part of the technical–chemical training of a student is the teaching laboratory in analytical chemistry. Analytical chemistry is a partial discipline that develops and applies methods, technologies and strategies in order to gain information on chemical compounds and processes. Principles of organic, inorganic and biological chemistry are supported by analytics, and the ever-increasing demand of society to make important decisions based on watertight data and validated methods requires the reinforcement of analytical education and research.

Following several years of being a teaching adviser in a university chemistry laboratory, I concerned myself with the validation of methods in environmental analytics in my capacity as an application chemist for a manufacturer of analytical equipment. In so doing I became aware of the different priorities placed on analytical processes in these various organizations.

In an academic environment the analytical chemistry laboratory serves, by way of experiments, to deepen what has been learned, to consolidate knowledge and to help apply it in a practical manner. The teaching laboratory complements the theoretical knowledge and understanding of analytical procedures learned during lectures. It serves to convey the knowledge and facilitate working techniques and practical skills through exercises. The lab courses are meant to consolidate the careful and independent execution of one's own experiments, to train the ability to observe, and to encourage professional scientific work. Nowadays, the reports written after each experiment are evaluated with regard to the formulation, validation and agreement of hypotheses. Often the basis for this is measurement results achieved in an academic environment. Perhaps even aspects of quality assurance are taken into consideration in the evaluation process. This all happens at educational institutions on a more or less voluntary basis and seldom is there the necessity to apply international guidelines or norms.

In analytical laboratories in chemical industry the quality management system consists of aspects of corporate policy, ergonomics, measurement technology, product assessment and electronic data processing, and is certified by quality audits. Such extensive quality assurance measures are conducted for ecological and economic reasons. The maintenance of product quality, and thus of competitiveness, obliges the companies to comply with extensive legal requirements. The work to be carried out within the framework of quality assurance is not only

necessary to guarantee correct and accurate analytical values, but can be absolutely necessary for Good Laboratory Practice (GLP)-compliant laboratories or laboratories that want to be accredited, and are, at the same time, critical for the survival of corporations.

After destiny led me back to the university, I decided to write this book to sensitize students to the issue of quality assurance. Analytical validation is a topic that has become increasingly important for practice over the past years as a result of increased efforts in quality assurance, and which will also continue to play an important role in the future. Validations are an integral part of analytical processes and are a part of the repertoire of chemists who work in analytics, such as study of the literature, experimental work and reporting. Reports of experiments run during lab classes are intended to illustrate theoretical explanations and have to contain experimental–methodological information about quality assurance measures. There is a growing need to take account of the statistical evaluation of the results. All validation steps, from the analytical question to the evaluation of the results, are involved in the analytical process of a lab experiment and described together with plausibility assessments. What is to be conveyed to the student is the fact that analysts, in their capacity as problem solvers, perform a service for certain groups of customers. Based on information supported by the strategy, structure and culture of the customer, analysts apply their techniques and broad knowledge. The results must be precise and exact to be used by the customers for their purposes. Students must be made aware of aspects such as personnel costs, equipment, methods and improvements of the analytical process achieved by rationalization, increased competition and customer satisfaction. The solution to the problem should in any case be processed in such a way as to be "fit for purpose". Moreover, analysts must be able to communicate clearly complex workflows and connections as well as results to customers in management, marketing or legal departments who are not versed in the subject.

There are several good books on analytical chemistry, but most of them go into such detail on the theory that the relationship to the practical implementation is diluted. The standard procedure for finding solutions to analytical problems outlined here should be a model for contemporary and practical work in analytical laboratories. It has been consciously kept general to be applicable to a wide diversity of problems and customer specifications. In the modern-day laboratory practice there is quite a series of variations and modifications of the procedural model presented in this book. However, everyone is familiar with the phased approach, from the order entry to the end of the project, which is also an essential part of the workflow presented in this book.

In view of the fact that approximately eighty percent of analysts will work in industry in future, I regard it as crucial to illustrate clearly to students the role of analytical chemists in the industrial environment. In this environment analysts are engaged as problem solvers and will be confronted with corporate requirements, such as time, costs and precision. Students will learn that there are no general values for the acceptance of measurement insecurity, selectivity or quality in an analytical process, but rather that the values must be defined prior to starting

the project. The results achieved must suffice with regard to the purpose and depend on the legal goals and limitations of the customer. The selection and degree of optimization of the methods, as well as the scope of validation and documentation, are reflected in the order and its execution.

This book is intended to serve as a practical support for students and as an aid to internship supervisors. It is meant as a guideline and attempts to be an understandable course book on the present-day status of practice. The trials run during laboratory classes and described here are formulated as projects and are meant to be carried out by the students in teamwork. In so doing, not only will specialist knowledge be deepened, but one will also learn to make decisions in a broader context. Analytical problems vary from trial to trial. However, the solution process is retained in the examples given during the internship and consists of the following:

- understanding of the chemistry and physics on which the analytical method is based
- knowledge of the analytical processes from the order entry to reporting
- awareness that analytical work is service

Wädenswil, Switzerland, October 2009 *Sergio Petrozzi*

## Preface to the English Edition

The German edition of this book aroused lively interest, and this has incited me to write an extended version for translation into English.

The range of analytical methods introduced has been substantially enlarged and the number of the laboratory experiments has also been extended. The description of the new experiments also follows the proven standard and each starts with a small introduction (real presentation of problems involving different analyses) to the background and a short introduction to the procedure chosen by the analyst.

Translated from the originally published edition in German under the title *Instrumentelle Analytik* by Mrs. Maria Schmitz.

In her capacity as a freelance lecturer for the English Department of the University of Applied Sciences in Mainz, Economic Faculty, Mrs. Schmitz lectures on Intercultural Competence – German and American Business Styles. In addition, she lectures in the Communication Skills Faculty of the private academy accadis Bildung.

Zürich, Switzerland, May 2012 *Sergio Petrozzi*

## Dedication

My sincere thanks go to Prof. Dr. Georg Schwedt for the foreword to this book and for giving me the opportunity to share his rich experience.

I owe a special thanks to Dr. Huldrych Egli (Büchi Labortechnik AG, Flawil/CH) for working out the project "*N*-Protein Determination according to Kjeldahl".

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- High-Performance Thin Layer Chromatography – HPTLC (CAMAG Laboratory, Muttenz/CH)
- Near-Infrared – NIR-Spectrometry (Büchi Labortechnik AG, Flawil/CH)
- Atomic Absorption Spectrometry – AAS (PerkinElmer AG, Schwerzenbach/CH)
- Karl Fischer Water Determination and Ion Chromatography (Metrohm Schweiz AG, Herisau/CH)
- *N*-Protein Determination according to Kjeldahl (Büchi Labortechnik AG, Flawil/CH)
- Field-Flow Fractionation – FFF (Postnova Analytics GmbH, Landsberg/DE)

Finally, I would like to thank everyone whom I have forgotten. Rest assured that this was not intentional.

## Foreword

Results from the application of instrumental analytical methods today often form the basis for political, legal and medical decisions that pertain not only to the recycling and maintenance of the quality of air, water, the earth or food, but also to the overall "quality of life". In the medical field, particularly biochemical analytics and pharmaceutical research depend on instrumental analytics. In the field of material sciences and in technical subjects, knowledge of the content of trace elements is, for example, an important prerequisite for ascertaining physical properties. Even such diverse sciences as geology, archeology and the restoration of works of art and antique books make use of analytical methods to solve the problems of their respective fields. In fact, there is hardly an area of experimental natural sciences that does not make use of chemical analytics in some form or another.

This makes it all the more important for students to become acquainted with the practice and problem orientation of instrumental analytics. That means they must learn to select a method suitable to a given task. Analytical procedure, in general, consists of several phases, ranging from sampling and sample preparation to the evaluation and assessment of the results of the analysis. In so doing, planning and assessing analytical data with regard to their reliability and accuracy (in other words the validation of an analytical procedure) plays a very important role. For students it is therefore necessary to learn not only the theoretical aspect of chosen analytical methods and applications, but also to practise how to perform the necessary steps to solve analytical tasks efficiently. This laboratory course book can be an essential contribution to such a procedure.

Bonn, Germany, June 2009 *Prof. Dr. Georg Schwedt*

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